Method of Test for

DETERMINATION OF RUTILE TITANIUM DIOXIDE IN WHITE WATERBORNE TRAFFIC PAINT VIA X-RAY DIFFRACTOMETER

DOTD Designation: TR 523-11

I. Scope

A. This method of test is designed to determine the amount of rutile titanium dioxide in white waterborne traffic paint by using an X-ray diffractometer.

B. Reference Documents:

1. ASTM D 1475 – Standard Test Method for Density of Liquid Coatings, Inks, and Related Products
2. ASTM D 2369 – Standard Test Method for Volatile Content of Coatings
3. ASTM D 3723 – Standard Test Method for Pigment Content of Water-Emulsion Paints by Low-Temperature Ashing
4. LA DOTD Sampling Procedure S 608

II. Apparatus

A. **X-Ray Diffractometer,** XRD
B. **Tin sheet**, 2 x 4 inches
C. **Metal Cutting Shears**
D. **Paper Towel**
E. **Personal Nuclear Gage Badge**
F. **Personal protective equipment** – eye protection and apron for handling paint.
G. **Waterborne traffic paint worksheet,** Figures 1a and 1b

III. Health Precautions

Wear eye protection and apron while handling paint. Maintain proper ventilation to prevent exposure to solvents when handling waterborne paints.

IV. Sample

Sample white waterborne traffic paint in accordance to LA DOTD Sampling Procedure S 608.

V. Procedure

1. Determine and record the total solids in accordance with ASTM D 2369 on waterborne traffic paint worksheet.
2. Determine and record the weight of the sample in accordance with ASTM D 1475 on waterborne traffic paint worksheet.
3. Dip the tin sheet into the paint, collecting a sample about ¾ the length of the tin.
4. Tap off excess paint and wipe back of tin clean with a paper towel.
5. Allow specimen to dry for at least 15 minutes.
6. Ensure the X-ray diffractometer is on and proceed with testing per manufacturer's instruction.
7. Determine and record the percent pigment (P) in accordance with ASTM D 3723 on waterborne traffic paint worksheet.

VI. Calculations

A. Refer to the rich text format, RTF, report (Figures 2a and 2b), which is generated from the X-Ray diffractometer, and collect the following height data: Silicone Dioxide (SO_2), Titanium Dioxide (TiO_2), and

Calcium Carbonate ($CaCO_3$). Calculate the total height of the peak positions of the compounds above to the nearest 0.01 counts (cts), using the following formula:

$$T_H = H_S + H_T + H_C$$

where,

H_S = height of Silicone Dioxide peak, cts
H_T = height of Titanium Dioxide peak, cts
H_C = height of Calcium Carbonate peak, cts
T_H = total height, cts

Note: *To identify the height of each component, use the following peak position ranges: Silicone Dioxide (26.2 to 26.8), Titanium Dioxide (27.1 to 27.7), and Calcium Carbonate (29.1 to 29.7).*

Example,

H_S = 105.44
H_T = 195.23
H_C = 1218.31

$$T_H = 105.44 + 195.23 + 1218.31$$
$$= 1518.98 \text{ cts}$$

B. Calculate the percent of Titanium Dioxide, to the nearest 0.01% using the following formula:

$$R = \frac{H_T}{T_H} \times 100$$

where,

H_T = height of Titanium Dioxide peak, cts
T_H = total height, cts
R = percent of Titanium Dioxide
100 = conversion factor for percentage

Example:

H_T = 195.23
T_H = 1518.98
100 = conversion factor for percentage

$$R = \frac{195.23}{1518.98} \times 100$$
$$= 0.1285 \times 100$$
$$= 12.85\,\%$$

C. Calculate the amount of TiO_2 to the nearest 0.01 lb/gal, using the following formula:

$$TiO_2 = \frac{R \times P \times W}{90 \times 100}$$

where,

R = percent of Titanium Dioxide
P = percent pigment (refer to ASTM D 3723)
W = weight of paint, per gallon (refer to ASTM D1475)
90 = purity of Titanium Dioxide
100 = conversion factor

Example,

R = 12.85
P = 61
W = 13.7

$$TiO_2 = \frac{12.85 \times 61 \times 13.7}{90 \times 100}$$
$$= \frac{10738.745}{9000}$$
$$= 1.2 \text{ lbs/gal}$$

VII. **Report**

Record the amount of titanium dioxide to the nearest 0.1 lb/gal on the waterborne traffic paint worksheet, LA DOTD 346.

VIII. **Normal Test Reporting Time**

Normal test reporting time is 2 days.

6/05

Louisiana Department of Transportation and Development
Materials & Testing Section
TRAFFIC PAINT

REMARKS 2

	TEST RESULTS (Max. of 15 characters)	P/F
	XXXXXXXXXXXXXXXXXXX	XXX
TYPE	287-005	XXX
COLOR (Clean, Bright, Untinted Binder-White; Fed. Test Standard #595 Color 13538-Yellow)	white	P
PH (ASTM E 70)	10.2	P
PIGMENT, % [(D-E)÷C] x 100 (ASTM D 3723)	61	P
(A) Wt. of Syringe + Sample, g		
(B) Wt. of Syringe, g		
(C) Wt. of Sample, g		
(D) Wt. of Foil + Sample After Furnace		
(E) Wt. of Foil		
	XXXXXXXXXXXXXXXXXXX	XXX
NON-VOLATILE IN VEHICLE, % (Fed. Test Method 141) [(%Solids - % Pigment) ÷ (100 - % Pigment)] x 100	44	P
TOTAL SOLIDS, % (B÷A) x 100 (ASTM D 2369)	78	P
Wt. of Sample + Syringe, g		
Wt. of Syringe, g		
Wt. of Sample, g (A)		
Wt. of Sample + Foil After Heat, g		
Wt. of Foil, g		
Wt. of Solids, g (B)		
	XXXXXXXXXXXXXXXXXXX	XXX
WEIGHT:	XXXXXXXXXXXXXXXXXXX	XXX
PER GALLON, LB (F) [(D-E) ÷10] (ASTM D 1475)	13.7	P
PER LITER, kg (F x 0.11983)	.	
Wt. of Cup + Sample, g (D)		
Wt. of Cup, g (E)		
Wt. of Sample, g (D-E)		
VISCOSITY (ASTM D 562)	85	P
Weight, g		
Time, Sec.		

09/ 061-001-00000 /346/ 22-737635 /N (OVER)

Figure 1a
Traffic Paint Worksheet (Front)

DRYING TIME , MINUTES (ASTM D 711)	10	P
DRY THROUGH, MINUTES (ASTM D 1640)		
INFRARED SPECTRA (TR 610)		P
X-RAY DIFFRACTION (TR 523)		P
FLEXIBILITY (Fed. Spec. TT-P-1952)	XXXXXXXXXXXXXXXXXX	
HIDING POWER @ .250 µm (Fed. Test 141 B)		
DRYING TO NO PICKUP, MINUTES (ASTM D 711)		
DAYLIGHT REFLECTANCE, %: (Fed. Test 141 B)	XXXXXXXXXXXXXXXXXX	XXX
WHITE	81	P
YELLOW		
FINENESS OF GRIND (ASTM D 1210)	4	P
FREEZE-THAW (ASTM D 2243)	XXXXXXXXXXXXXXXXXX	
HEAT STABILITY (Fed. Spec. TT-P-1952)	XXXXXXXXXXXXXXXXXX	
VOLATILE ORGANIC COMPOUNDS, g/L		
PIGMENT COMPOSITION:	XXXXXXXXXXXXXXXXXX	XXX
WHITE PAINT: (ASTM D 476)	XXXXXXXXXXXXXXXXXX	XXX
TITANIUM DIOXIDE WEIGHT, Weight, lb/gal (A)	11.2	P
g/L (A x 119.83)		

Tested by: MC Date: 1/5/10 Checked by: AT Date: 1/8/10

APPROVED by: _____ Date: _____

Figure 1b
Traffic Paint Worksheet (Back)

Date: 1/7/2010 Time: 11:10:11 AM File: 737635.rd User: pcuser

Document History

Insert Measurement:
- Scan name = "737635.rd"
- Modification time = "1/6/2010 10:23:45 AM"
- Modification editor = "pcuser"

Search Peaks:
- Minimum significance = "3.00"
- Minimum tip width = "0.01"
- Maximum tip width = "1.00"
- Peak base width = "2.00"
- Method = "Minimum 2nd derivative"
- Modification time = "1/6/2010 10:23:51 AM"
- Modification editor = "pcuser"

Peak List

	Pos.[°2Th.]	Height[cts]	FWHM[°2Th.]	d-spacing[Å]	Rel.Int.[%]
	23.1634	128.06	0.1574	3.83999	10.51
SiO_2	26.7454	105.44	0.1181	3.33329	8.65
TiO_2	27.4977	195.23	0.1574	3.24378	16.02
$CaCO_3$	29.5302	1218.31	0.1968	3.02498	100.00

$$\frac{195.23}{1518.98} \times 100 = 12.85\%\ TiO_2$$

Figure 2a
X-Ray Diffractometer RTF Report

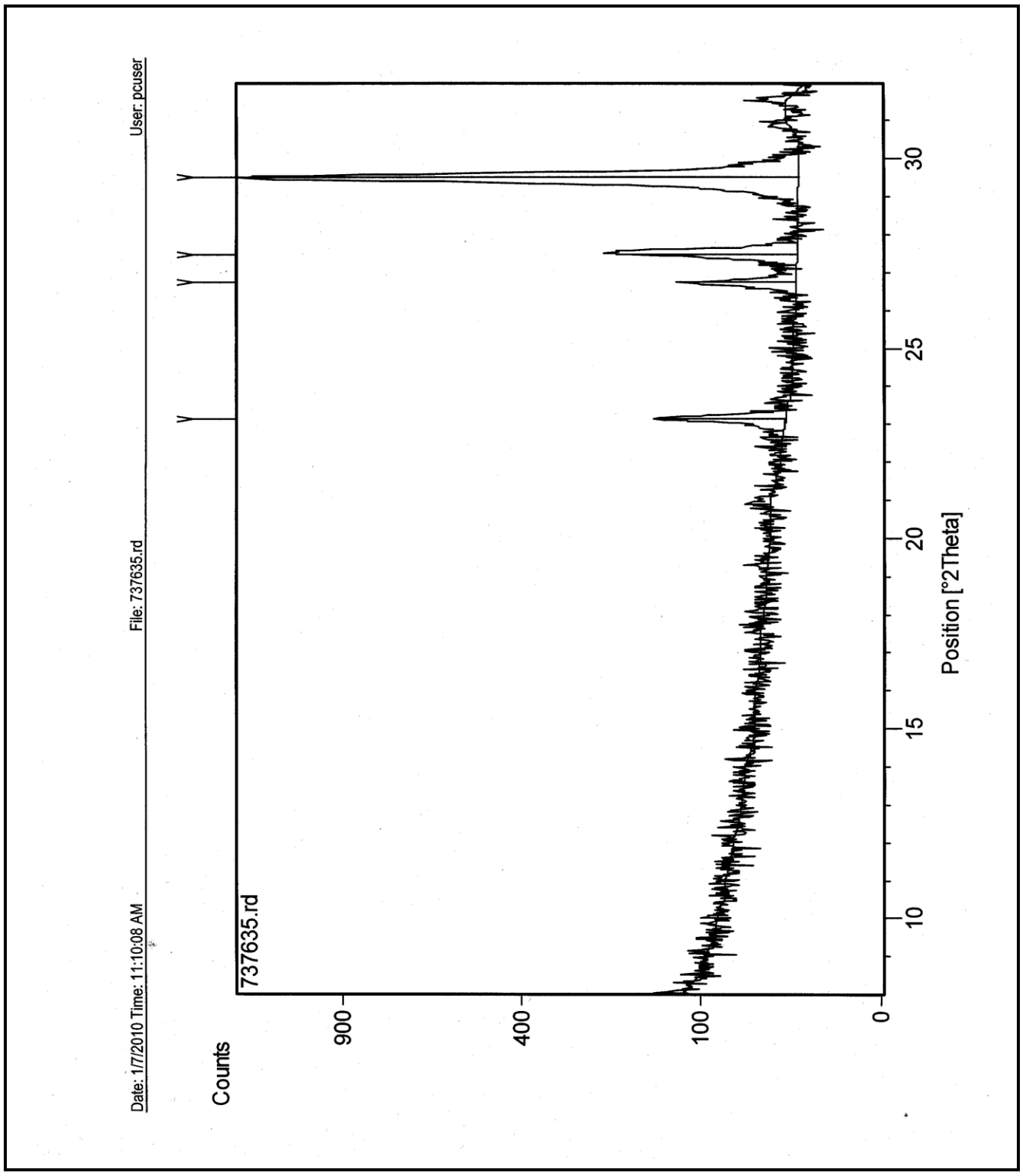

Figure 2b
X-Ray Diffractometer RTF Report